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(54) Title: NON-POSTDRAWN POLYOLEFIN FIBERS WITH HIGH TENACITY

(57) Abstract: The invention relates to non-postdrawn polyolefin fibers with high tenacity values of 2,0 to 5,0 cN/dtex, consisting essentially of predominantly isotactic propylene polymers having a crystallisation temperature of < 116 °C (DSC) and 0,001 to 2 % by weight, based on the propylene polymers used, of  $\alpha$ -nucleating agents a process for producing them and their use.

### Non-postdrawn polyolefin fibers with high tenacity

The invention relates to non-postdrawn polyolefin fibers with high tenacity from propylene polymers as well as to a process for producing them.

Fibers from propylene polymers are known (Ullmann's Encyclopedia of Industrial Chemistry, Vol. A10, p. 536-542, Weinheim 1987).

Known methods for producing high tenacity fibers are oxidative chain scission degradation of hot spun fibers in conjunction with a delayed quench step (U.S. 5 281 378, US 5 318 735), the use of reactor-grade propylene polymers produced by single-site catalysis (EP 0 854 212) for producing the fibers, or the use of blends consisting of mixtures of propylene homopolymers, propylene copolymers and heterophasic propylene polymers being stabilized with organic phosphites, phenolic antioxidants or HALS-compounds for producing the fibers (EP 0 632 147). The disadvantage of these methods is, that non-postdrawn propylene polymer fibers having tenacity values of 2,0 to 5,0 cN/dtex cannot be realised.

It is the object of the present invention to provide non-postdrawn polyolefin fibers having tenacity values of 2,0 to 5,0 cN/dtex comprising propylene polymers.

According to the present invention, this object is achieved by non-postdrawn polyolefin fibers consisting essentially of predominantly isotactic propylene polymers having a crystallisation temperature of  $> 116^{\circ}\text{C}$  (DSC) and 0,001 to 2 % by weight, based on the propylene polymers used, of  $\alpha$ -nucleating agents which are selected from the group consisting of

polymers which are different from isotactic propylene polymers, which are selected from the group consisting of

tetrafluoroethylene polymers, polycarbonates, polybutyleneterephthalate, polyethyleneterephthalate, 3-methylbutene polymers, 4-methylpentene-1-polymers, syndiotactic propylene polymers, polyphenyleneoxides, propylene-methylbutene copolymers, styrene-acrylonitrile copolymers, polyallyltrimethylsilanes and hydrolysed ethylene vinylacetate copolymers,

and benzoic acid, benzoic acid derivatives, dibenzylidene sorbitol, derivatives of sorbitol, diphenylglycine, salts of C<sub>6</sub>-C<sub>18</sub> aliphatic carbonic acids, salts of C<sub>7</sub>-C<sub>13</sub> aromatic carbonic acids, phosphoric acid derivatives, talc and mixtures thereof.

The predominantly isotactic propylene polymers are essentially consisting of

- (a) 50 – 100 wt% propylene polymers, preferably propylene homopolymers, produced using Ziegler-Natta catalysts or metallocene catalysts, especially with an Mw/Mn ratio of 2 to 4.5, and/or copolymers of propylene, ethylene and/or  $\alpha$ -olefins with 4 to 18 carbon atoms and a propylene content of 80.0 to 99.9 % by weight in the form of random copolymers, block copolymers and/or random block copolymers, with melt indexes of 0.5 to 40 g/10 min at 230 °C/2.16 kg and preferably of 1 to 5 g/10 min at 230 °C/2.16 kg,
- (b) 0 - 50 wt % of a polyolefin mixture with an Mw/Mn ratio of 2 to 6 and a melt index of 0.5 to 40 g/10 min at 230 °C/2.16 kg, essentially consisting of
  - (b1) 60 to 98 % by weight of a crystalline copolymer of 85 to 95 % by weight of propylene and 15 to 0.5 % by weight of ethylene and/or an  $\alpha$ -olefin of the general formula  $\text{CH}_2 = \text{CHR}$ , wherein R is a linear or branched alkyl group with 2 to 8 carbon atoms,
  - (b2) 2 to 40 % by weight of an elastic copolymer of 20 to 70 % by weight of ethylene and 80 to 30 % by weight of propylene and/or an  $\alpha$ -olefin of the general formula  $\text{CH}_2\text{CHR}$ , wherein R is a linear or branched alkyl group with 2 to 8 carbon atoms,
- (c) 0 – 30 wt% of highly amorphous polypropylenes or propylene copolymers with a crystalline poly-propylene or crystalline propylene copolymer content of less than 10 % by weight, an enthalpy of melting of less than 40 J/g and a melt index of 0.5 to 40 g/10 min at 230 °C/2.16 kg, the largely amorphous polypropylene being a homopolymer of propylene and/or a copolymer of propylene of at least 80 mole percent propylene and not more than 20 mol percent of one or more  $\alpha$ -olefins of the general formula  $\text{CH}_2\text{CHR}$ , wherein R is a linear or branched alkyl group with 2 to 8 carbon atoms,

- (d) 0 – 10 wt% of non-isotactic propylene homopolymers with a melting point of 145° to 165 °C, a melt viscosity of 200,000 cps at 190 °C and a heat of crystallization of 4 to 10 calories per gram, 35 to 55 % by weight being soluble in diethyl ether,
- (e) 0 - 30 wt% of modified propylene homopolymers and/or propylene copolymers with melt indexes of 0.5 to 40 g/10 min at 230 °C/2.16 kg and preferably of 2 to 5 g/10 min at 230 °C/2.16 kg, and a ratio of the intrinsic viscosity of the modified propylene polymer to the intrinsic viscosity of the basic unmodified polymer (component a) with largely the same weight average molecular weight of 0,2 to 0,95.

The polyolefin mixtures b) of crystalline copolymers and elastic copolymers, optionally contained in the non-postdrawn polyolefin fibers, are polymer mixtures described, for example, in the European patents 0 400 333 or 0 472 946.

The largely amorphous polypropylenes or propylene copolymers c), optionally contained in the non-postdrawn polyolefin fibers are, in particular, stereo block polypropylenes, which are prepared, for example, by using highly active Ziegler-Natta catalysts fixed on a metal oxide (Collette, J., *Macromolecules* 22 (1989), 3851 - 3858; German patent 28 30 160) or soluble Ziegler-Natta catalysts (de Candia, F., *Makromol. Chem.* 189 (1988), 815 - 821), optionally with subsequent reactive modification (European patent 636863) and/or degradation (European patent 640 850).

Examples for the non-isotactic propylene homopolymers d), optionally contained in the non-postdrawn polyolefin fibers are the products described p.e. in the European patent 0 475 307 or in the European patent 0 475 308.

The modified propylene polymers e), optionally contained in the non-postdrawn polyolefin fibers, can be produced by any number of processes, e.g. by treatment of the unmodified propylene polymer with thermal decomposing radical-forming agents and/or by treatment with ionizing radiation, where both treatments may optionally be accompanied or followed by a treatment with bi- or multifunctionally unsaturated monomers, e.g. butadiene, isoprene, dimethylbutadiene or divinylbenzene. Further processes may be suitable for the production of the modified propylene polymer, provided that the resulting modified propylene polymer meets the characteristics given above.

Examples for said modified propylene polymers e) optionally contained in the non-

postdrawn polyolefin fibers, are, in particular :

- polypropylenes modified by the reaction of polypropylenes with bismaleimido compounds in the melt (EP-A-0 574 801; EP-A-0 574 804),
- polypropylenes modified by the treatment of polypropylenes with ionizing radiation in the solid phase (EP-A-0 190 889; EP-A-0 634 454),
- polypropylenes modified by the treatment of polypropylenes with peroxides in the solid phase (EP-A-0-384 431) or in the melt (EP-A-0-142724),
- polypropylenes modified by the treatment of polypropylenes with multifunctional, ethylenically unsaturated monomers using ionizing radiation (EP-A-0 678 527)
- polypropylenes modified by the treatment of polypropylenes with multifunctional, ethylenically unsaturated monomers in the presence of peroxides in the melt (EP-A-0 688 817; EP-A-0 450 342)

These modified polypropylenes have melt indexes of 0.5 to 40 g/10 min at 230 °C/2.16 kg and preferably of 2 to 5 g/10 min at 230 °C/2.16 kg, and a ratio of the intrinsic viscosity of the modified propylene polymer to the intrinsic viscosity of the basic unmodified polymer (component a) with largely the same weight average molecular weight of 0,2 to 0,95.

These modified polypropylene polymers normally exhibit strain hardening behaviour as usually defined in the literature.

Examples for tetrafluoroethylene polymers, optionally contained in the non-postdrawn poly-olefin fibers, are polytetrafluoroethylene, tetrafluoroethylene propylene copolymers and tetrafluoroethylene isobutylene copolymers.

Examples for polycarbonates, optionally contained in the non-postdrawn polyolefin fibers, are poly(4,4'-isopropylidenediphenylene carbonate) and copolycondensates based on 4,4'-isopropylidenebisphenol, 4,4'-tetrabromoisopropylidenebisphenol and/or 4,4'-hexabromoiso-propylidenebisphenol.

Examples for 3-methylbutene polymers, optionally contained in the non-postdrawn poly-olefin fibers, are poly-3-methylbutene, 3-methylbutene 1-butene copolymers or 3-methylbutene isobutylene copolymers.

Examples for 4-methylpentene-1-polymers, optionally contained in the non-postdrawn poly-olefin fibers, are poly-4-methylpentene-1, 4-methylpentene-1 styrene copolymers or 4-methylpentene-1 propylene copolymers.

Examples for polyphenyleneoxides, optionally contained in the non-postdrawn polyolefin fibers, are poly-2,6-dimethylphenyleneoxide, poly-2,6-diisopropylphenyleneoxide or poly-2,6-diphenylphenyleneoxide.

Examples for  $C_7$ - $C_{13}$  benzoic acid derivatives, optionally contained in the non-postdrawn polyolefin fibers, are benzoic anhydride, o-toluic acid and 2,6-dimethylbenzoic acid.

Examples for sorbitol derivatives, optionally contained in the non-postdrawn polyolefin fibers, are di-(p-methylbenzylidene) sorbitol, trinaphthylidenesorbitol and naphthylmethylenesorbitol.

Examples for salts of  $C_7$ - $C_{13}$  aromatic carbonic acids are sodium benzoate and tertiary-butylbenzoic acid aluminium salt.

A preferred aromatic carbonic acid is sodium benzoate.

Examples for phosphoric acid derivatives are ammonium polyphosphate, cyclic calcium phosphate compounds, sodium-2,2'-methylenbis-(4,6-di-tert.-butylphenyl)phosphate and bis-(tertiary-butyl) phosphoric acid sodium salt.

A preferred cyclic calcium phosphate compound, optionally contained in the non-postdrawn polyolefin fibers, is bis(2,6-di-tert.butyl-1-hydroxy)methane calcium phosphate.

A further  $\alpha$ -nucleation method, which is also suitable, is a special reactor technique, where the catalyst is prepolymerised with monomers like vinylcyclohexane (VCH). This method is described in greater detail in e.g. EP 0 316 187 A2.

In addition to the nucleating agents mentioned above, the use of long-chain branched polypropylene with high melt strength for nucleation purposes as outlined e.g. in US 6,077,907 is included.

Preferred auxiliary materials are 0.01 to 2.5 % by weight of stabilizers and/or 0.01 to 1 % by weight of processing aids and/or, optionally, 0.1 to 1 % by weight of antistatic agents

and/or 0.2 to 3 % by weight of pigments and/or 2 to 20 % by weight of flame retardants, in each case based on the sum of the polypropylenes.

The stabilizers, contained in the inventive non-postdrawn polyolefin fibers, preferably are mixtures of 0.01 % to 0.6 % by weight of phenolic antioxidants, 0.01 % to 0.6 % by weight of 3-arylbenzofuranones, 0.01 % to 0.6 % by weight of processing stabilizers based on phosphites, 0.01 % to 0.6 % by weight of high temperature stabilizers based on disulfides and thioethers and/or 0.01 % to 0.8 % by weight of sterically hindered amines (HALS).

Suitable phenolic antioxidants are 2-t-butyl-4,6-dimethylphenol, 2,6-di-t-butyl-4-methylphenol, 2,6-di-t-butyl-4-isoamylphenol, 2,6-di-t-butyl-4-ethylphenol, 2-t-butyl-4,6-diisopropylphenol, 2,6-dicyclopentyl-4-methylphenol, 2,6-di-t-butyl-4-methoxymethylphenol, 2-t-butyl-4,6-dioctadecylphenol, 2,5-di-t-butylhydroquinone, 2,6-di-t-butyl-4,4-hexadecyloxyphenol, 2,2'-methylene-bis(6-t-butyl-4-methylphenol), 4,4'-thio-bis-(6-t-butyl-2-methylphenol), octadecyl 3(3,5-di-t-butyl-4-hydroxyphenyl) propionate, 1,3,5-trimethyl-2,4,6-tris(3',5'-di-t-butyl-4-hydroxybenzyl)benzene and/or pentaerythritol-tetrakis-3-(3,5-di-t-butyl-4-hydroxyphenyl)-propionate.

As benzofuranone derivative, 5,7-di-t-butyl-3-(3,4-dimethylphenyl)-3H-benzofuran-2-one, in particular, is suitable.

As HALS compounds, bis-2,2,6,6-tetramethyl-4-piperidyl sebacate and/or poly-1,1,3,3-tetra-methylbutyl-imino-1,3,5-triazine-2,4-diyl(2,2,6,6-tetramethylpiperidyl)-amino-hexamethylene-4-(2,2,6,6-tetramethylpiperidyl)-imino are particularly suitable.

As processing aids, the inventive non-postdrawn polyolefin fibers may contain calcium stearate, magnesium stearate and/or waxes.

According to an advantageous embodiment of the present invention the predominantly isotactic propylene polymers comprise from 5 - 100 ppm of particles of inorganic oxides having a particle diameter from about 0.05 - 10  $\mu\text{m}$ .

Inorganic oxides selected from the group consisting of ZnO, SnO, MgO,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{TiO}_2$ ,  $\text{ZrO}_2$ ,  $\text{HfO}_2$  and mixtures thereof in amounts of from 20 - 40 ppm are preferred.

Still more preferred are particles of inorganic oxides of  $\text{Al}_2\text{O}_3$  and/or  $\text{SiO}_2$  and/or  $\text{MgO}$  having a particle diameter from about 0.5 to 2.0  $\mu\text{m}$ .

A still further object of the present invention is a process for preparing the inventive non-postdrawn polyolefin fibers having tenacity values of 2,0 to 5,0 cN/dtex, characterized by melting of predominantly isotactic propylene polymers having a crystallisation temperature of  $> 116^\circ\text{C}$  (DSC) and 0,001 to 2 % by weight, based on the propylene polymers used, of  $\alpha$ -nucleating agents which are selected from the group consisting of

polymers which are different from isotactic propylene polymers, which are selected from the group consisting of

tetrafluoroethylene polymers, polycarbonates, polybutyleneterephthalate, polyethyleneterephthalate, 3-methylbutene polymers, 4-methylpentene-1-polymers, syndiotactic propylene polymers, polyphenyleneoxides, propylene-methylbutene copolymers, styrene-acrylonitrile copolymers, polyallyltrimethylsilanes and hydrolysed ethylene vinylacetate copolymers, and benzoic acid, benzoic acid derivatives, dibenzylidene sorbitol, derivatives of sorbitol, diphenylglycine, salts of  $\text{C}_6\text{-C}_{18}$  aliphatic carbonic acids, salts of  $\text{C}_7\text{-C}_{13}$  aromatic carbonic acids, phosphoric acid derivatives, talc and mixtures thereof,

at temperatures of about 220 to 325  $^\circ\text{C}$ , conveying the melt to spinnerets, extruding the melt into fibers and subsequently cooling the fibers and taking up the resulting hot spun fibers at speeds of 1000 – 6000 m/min..

As plasticising extruder for melting the mixtures, especially single screw extruders or twin screw extruders with screw length of 28 to 30 D, preferably with flange-mounted static or dynamic mixers, are suitable. Shear speeds can be adjusted to values of  $10^2$  /sec to  $10^3$ /sec by controlling the temperature and the rpm.

For uniformly metering the mixtures, which has been melted in the plasticizing extruder, over the melt distributor to the capillary die, melt pumps, preferably heated with biphenyl, are used for the melts, heated to 240° to 310  $^\circ\text{C}$ .



According to a feature of the present invention, the resulting hot spun fibers are taken up by high speed gallettes, cabling the fibers into multifilament yarns and winding up the non-postdrawn polyolefin fibers-containing yarns.

Preferred applications of the non-postdrawn polyolefin fibers are the manufacturing of nonwovens, preferred spunbond, carded or air bonded nonwovens; textile applications, carpets.

### Examples

For the production of polyolefin fibers, which are not drawn subsequently, the inventive method is explained, by way of example, by a method outlined in Fig. 1. In Fig. 1, 1 is the extruder, 2 the extrusion pump, 3 the spinneret, 4 the blast shaft, 5 the pull-off equipment and 6 the winder.

As extruder (1) for melting the polyolefin mixtures, a single screw extruder is used with a high homogenizing effect with a screw length of 34 D and a flange-mounted static mixer. The spinneret (3) has an internal diameter of 0.5 mm. In the pull-off equipment (5), the pulling-off can be accomplished directly by means of the winder (6). Possible pull-off speeds are 1000 to 6000 m/min.

The melt indices for propylene polymers were measured with a load of 2.16 kg at 230 °C. The melt index is that quantity of polymer in grams which the test apparatus standardized to DIN 53 735 extrudes within 10 minutes at a temperature of 230 °C under a weight of 2.16 kg.

Tenacity and tensile elongation are both determined according to DIN 53834/1.

### Example 1

For producing a polyolefin polymer blend for high speed melt spinning, a dry mixture consisting of 85 % by weight of a metallocene propylene homopolymer (melt index of 6.2 g/10 minutes at 230 °C/2.16 kg), 13 % by weight of a propylene ethylene copolymer (melt index of 4.2 g/10 minutes at 230 °C/2.16 kg, ethylene content 4.8 % by weight), 2 % by weight of a 4-methylpentene-1 propylene copolymer (melt index of 0.25 g/10 minutes at 230 °C/5 kg, propylene content 6.8 % by weight), and as adjuvants, the percentages in each case based on the sum of the propylene polymers, 0.25 % by weight of 2-t-butyl-4,6-diisopropylphenol, 0.2 % by weight of bis-2,2,6,6-tetramethyl-4-piperidyl sebacate and 0.2 % by weight of calcium stearate, is melt blended at a melt temperature of 230 °C in a twin screw extruder and granulated.

In the spinning equipment of Figure 1, the blend is melted in the extruder at a mass temperature of 275 °C. The melt is transferred by the extrusion pump to the spinneret and, at a spinneret temperature of 290 °C, drawn off through the blast shaft, which is cooled with compressed air at a temperature of 20 °C, and wound up at a speed of 4000 m/min.

The resulting polypropylene fiber, which is not postdrawn, has a tenacity of 3.6 cN/dtex and a tensile elongation of 105 %.

### Example 2

For producing a polyolefin polymer blend for high speed melt spinning, a dry mixture consisting of 90 % by weight of a metallocene propylene homopolymer (melt index of 6.2 g/10 minutes at 230 °C/2.16 kg), 5 % by weight of a random heterophasic propylene ethylene blockcopolymer (melt index of 8 g/10 minutes at 230 °C/2.16 kg, ethylene content 33 mol%) 5 % by weight of a propylene ethylene copolymer (melt index of 4,2 g/10 minutes at 230 °C/2.16 kg, ethylene content 4,8 % by weight) and, the percentages in each case based on the sum of the propylene polymers, 0,2 % by weight of dibenzylidene sorbitol, 0.25 % by weight of 3(3,5-di-t-butyl-4-hydroxyphenyl)propionate, 0.2 % by weight of bis-2,2,6,6-tetra-methyl-4-piperidyl sebacate and 0.2 % by weight of magnesium stearate, is melt blended at a melt temperature of 220 °C in a twin screw extruder and granulated.

In the spinning equipment of Figure 1, the blend is melted in the extruder at a mass temperature of 280 °C. The melt is transferred by the extrusion pump to the spinneret and, at a spinneret temperature of 285 °C, drawn off through the blast shaft, which is cooled with compressed air at a temperature of 20 °C, and wound up at a speed of 3500 m/min.

The resulting polypropylene fiber, which is not postdrawn, has a tenacity of 3.2 cN/dtex and a tensile elongation of 122 %.

### Example 3

For producing a polyolefin polymer blend for high speed melt spinning, a dry mixture consisting of 85 % by weight of a metallocene propylene homopolymer (melt index of 6.2 g/10 minutes at 230 °C/2.16 kg), 5 % of a propylene homopolymer having strain

hardening behavior (melt index of 3,5 g/10 minutes at 230 °C/2.16 kg, strain hardening behaviour as usually defined in the literature), 10 % by weight of a Ziegler-Natta propylene homopolymer with high stereospecificity (melt index of 2,5 g/10 minutes at 230 °C/2.16 kg), and, the percentages in each case based on the sum of the propylene polymers, 0,35 % by weight of sodium-2,2'-methylenebis-(4,6-di-tert.-butylphenyl)phosphate, 0.25 % by weight of 2-t-butyl-4,6-diisopropylphenol, 0.2 % by weight of bis-2,2,6,6-tetramethyl-4-piperidyl seba-cate and 0.2 % by weight of calcium stearate, is melt blended at a melt temperature of 225 °C in a twin screw extruder and granulated.

In the spinning equipment of Figure 1, the blend is melted in the extruder at a mass temperature of 285 °C. The melt is transferred by the extrusion pump to the spinneret and, at a spinneret temperature of 290 °C, drawn off through the blast shaft, which is cooled with compressed air at a temperature of 20 °C, and wound up at a speed of 3000 m/min.

The resulting polypropylene fiber, which is not postdrawn, has a tenacity of 3.0 cN/dtex and a tensile elongation of 137 %.

## Claims

1. Non-postdrawn polyolefin fibers with high tenacity values of 2,0 to 5,0 cN/dtex, consisting essentially of predominantly isotactic propylene polymers having a crystallisation temperature of  $> 116^{\circ}\text{C}$  (DSC) and 0,001 to 2 % by weight, based on the propylene polymers used, of  $\alpha$ -nucleating agents which are selected from the group consisting of  
polymers which are different from isotactic propylene polymers, which are selected from the group consisting of  
tetrafluoroethylene polymers, polycarbonates, polybutyleneterephthalate, polyethyleneterephthalate, 3-methylbutene polymers, 4-methylpentene-1-polymers, syndiotactic propylene polymers, polyphenyleneoxides, propylene-methylbutene copolymers, styrene-acrylonitrile copolymers, polyallyltrimethylsilanes and hydrolysed ethylene vinylacetate copolymers, and benzoic acid, benzoic acid derivatives, dibenzylidene sorbitol, derivatives of sorbitol, diphenylglycine, salts of  $\text{C}_6\text{-C}_{18}$  aliphatic carbonic acids, salts of  $\text{C}_7\text{-C}_{13}$  aromatic carbonic acids, phosphoric acid derivatives, talc and mixtures thereof.
2. Non-postdrawn polyolefin fibers as claimed in claim 1, wherein said predominantly isotactic propylene polymers are essentially consisting of
  - (c) 50 – 100 wt% propylene polymers, preferably propylene homopolymers, produced using Ziegler-Natta catalysts or metallocene catalysts, especially with an Mw/Mn ratio of 2 to 4.5, and/or copolymers of propylene, ethylene and/or  $\alpha$ -olefins with 4 to 18 carbon atoms and a propylene content of 80.0 to 99.9 % by weight in the form of random copolymers, block copolymers and/or random block copolymers, with melt indexes of 0.5 to 40 g/10 min at  $230^{\circ}\text{C}/2.16\text{ kg}$  and preferably of 1 to 5 g/10 min at  $230^{\circ}\text{C}/2.16\text{ kg}$ ,
  - (d) 0 - 50 wt % of a polyolefin mixture with an Mw/Mn ratio of 2 to 6 and a melt index of 0,5 to 40 g/10 min at  $230^{\circ}\text{C}/2.16\text{ kg}$ , essentially consisting of
    - (b1) 60 to 98 % by weight of a crystalline copolymer of 85 to 95 % by weight of propylene and 15 to 0.5 % by weight of ethylene and/or an  $\alpha$ -olefin of the general formula  $\text{CH}_2 = \text{CHR}$ , wherein R is a linear or branched alkyl group with 2 to 8 carbon atoms,

- (b2) 2 to 40 % by weight of an elastic copolymer of 20 to 70 % by weight of ethylene and 80 to 30 % by weight of propylene and/or an  $\alpha$ -olefin of the general formula  $\text{CH}_2\text{CHR}$ , wherein R is a linear or branched alkyl group with 2 to 8 carbon atoms,
- (f) 0 – 30 wt% of highly amorphous polypropylenes or propylene copolymers with a crystalline poly-propylene or crystalline propylene copolymer content of less than 10 % by weight, an enthalpy of melting of less than 40 J/g and a melt index of 0.5 to 40 g/10 min at 230 °C/2.16 kg, the largely amorphous polypropylene being a homopolymer of propylene and/or a copolymer of propylene of at least 80 mole percent propylene and not more than 20 mol percent of one or more  $\alpha$ -olefins of the general formula  $\text{CH}_2\text{CHR}$ , wherein R is a linear or branched alkyl group with 2 to 8 carbon atoms,
- (g) 0 – 10 wt% of non-isotactic propylene homopolymers with a melting point of 145° to 165 °C, a melt viscosity of 200,000 cps at 190 °C and a heat of crystallization of 4 to 10 calories per gram, 35 to 55 % by weight being soluble in diethyl ether,
- (h) 0 - 30 wt% of modified propylene homopolymers and/or propylene copolymers with melt indexes of 0.5 to 40 g/10 min at 230 °C/2.16 kg and preferably of 2 to 5 g/10 min at 230 °C/2.16 kg, and a ratio of the intrinsic viscosity of the modified propylene polymer to the intrinsic viscosity of the basic unmodified polymer (component a) with largely the same weight average molecular weight of 0,2 to 0,95.
3. Non-postdrawn polyolefin fibers as claimed in claim 1 or 2, further comprising conventional additives.
4. Non postdrawn polyolefin fibers as claimed in claim 3, wherein said conventional additives are 0.01 to 2.5 % by weight of stabilizers, 0.01 to 1 % by weight of processing aids and, optionally, 0. 1 to 1 % by weight of antistatic agents, 0.2 to 3 % by weight of pigments and 2 to 20 % by weight of flame retardants, in each case based on the sum of the polypropylenes.
5. Non-postdrawn polyolefin fibers according to any one of claims 1 to 4 characterized in that the predominantly isotactic propylene polymers comprise from 5 - 100 ppm of particles of inorganic oxides having a particle diameter from about 0.05 - 10  $\mu\text{m}$ .

6. Non-postdrawn polyolefin fibers according to claim 5, characterized in that the predominantly isotactic propylene polymers comprise from 20 -40 ppm of inorganic particles selected from the group consisting of ZnO, SnO, MgO, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, TiO<sub>2</sub>, ZrO<sub>2</sub>, HfO<sub>2</sub> and mixtures thereof.

7. Non-postdrawn polyolefin fibers according to one of claims 5 or 6, characterized in that the particles of inorganic oxides are Al<sub>2</sub>O<sub>3</sub> and/or SiO<sub>2</sub> and/or MgO having a particle diameter from about 0.5 to 2.0  $\mu$ m.

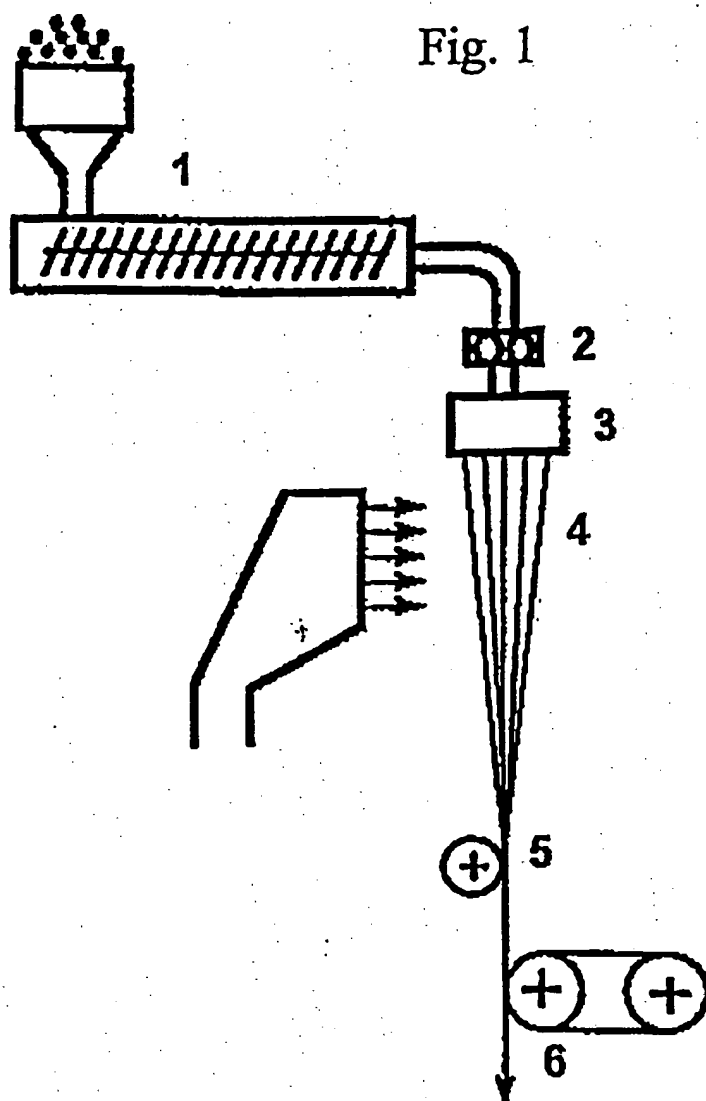
8. Process for preparing non-postdrawn polyolefin fibers according to any one of claims 1 to 7, characterised by melting of predominantly isotactic propylene polymers according to any one of claims 1 - 7 and 0,001 to 2 % by weight, based on the propylene polymers used of  $\alpha$ -nucleating agents according to claim 1 at temperatures of about 220 to 325 °C, conveying the melt to spinnerets, extruding the melt into fibers and subsequently cooling the fibers and taking up the resulting hot spun fibers at speeds of 1000 – 6000 m/min.

9. Process for preparing non-postdrawn polyolefin fibers as claimed in claim 8, characterised in that the resulting hot spun fiber is taken up by high speed gallettes, the fibers are cabled into multifilament yarns and the resulting yarns containing the non-postdrawn polyolefin fibers are wound up.

10. Use of non-postdrawn polyolefin fibers of any one of the claims 1 to 7 for manufacturing of nonwovens, preferred spunbond, carded or air bonded nonwovens; for textile applications, and carpets.

1/1

Fig. 1





## INTERNATIONAL SEARCH REPORT

 Int. Application No.  
 PCT/EP 01/14155

 A. CLASSIFICATION OF SUBJECT MATTER  
 IPC 7 D01F6/06 D01F6/46 D01F1/10

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

 Minimum documentation searched (classification system followed by classification symbols)  
 IPC 7 D01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the International search (name of data base and, where practical, search terms used)

EP0-Internal, WPI Data, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 878 567 A (PCD POLYMERE AG) 18 November 1998 (1998-11-18) the whole document	1-10
Y	WO 99 24478 A (BOREALIS AS ; JAEAESKELAEINEN PIRJO (FI); MALM BO (FI); KARBASI AMI) 20 May 1999 (1999-05-20) the whole document	1-10
Y	WO 99 24479 A (BOREALIS AS ; JAEAESKELAEINEN PIRJO (FI); MALM BO (FI); HUOVINEN PA) 20 May 1999 (1999-05-20) the whole document	1-10
Y	EP 0 854 155 A (JAPAN POLYOLEFINS CO LTD) 22 July 1998 (1998-07-22) the whole document	1-10
	-/-	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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- \*P\* document published prior to the international filing date but later than the priority date claimed

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- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- \*G\* document member of the same patent family

Date of the actual completion of the International search

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# INTERNATIONAL SEARCH REPORT

In 1st Application No  
PCT/EP 01/14155

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 790 262 A (CHISSO CORP) 20 August 1997 (1997-08-20) page 8, line 50 -page 19, line 26; claims 1,6-9	1-10
Y	EP 0 569 860 A (KIMBERLY CLARK CO) 18 November 1993 (1993-11-18) the whole document	1-10
Y	US 5 137 973 A (KHANNA YASH P ET AL) 11 August 1992 (1992-08-11) the whole document	1-10

## INTERNATIONAL SEARCH REPORT

In 1st Application No  
PCT/EP 01/14155

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
EP 0878567	A	18-11-1998	DE 19720135 A1	19-11-1998
			DE 19722579 A1	03-12-1998
			CZ 9801437 A3	12-05-1999
			EP 0878567 A2	18-11-1998
			US 6218011 B1	17-04-2001
			US 2002002241 A1	03-01-2002
WO 9924478	A	20-05-1999	FI 974175 A	08-05-1999
			AT 203548 T	15-08-2001
			AU 744410 B2	21-02-2002
			AU 1035699 A	31-05-1999
			BR 9813986 A	26-09-2000
			CA 2308076 A1	20-05-1999
			CN 1285850 T	28-02-2001
			DE 69801236 D1	30-08-2001
			EP 1028984 A1	23-08-2000
			ES 2162471 T3	16-12-2001
			WO 9924478 A1	20-05-1999
			JP 2001522903 T	20-11-2001
			PL 340376 A1	29-01-2001
WO 9924479	A	20-05-1999	AU 1035799 A	31-05-1999
			BR 9814854 A	03-10-2000
			CA 2308075 A1	20-05-1999
			CN 1285851 T	28-02-2001
			EP 1028985 A1	23-08-2000
			WO 9924479 A1	20-05-1999
			JP 2001522904 T	20-11-2001
EP 0854155	A	22-07-1998	EP 0854155 A1	22-07-1998
			US 6162887 A	19-12-2000
			WO 9804600 A1	05-02-1998
EP 0790262	A	20-08-1997	AU 717990 B2	06-04-2000
			AU 6838296 A	19-03-1997
			DE 69608240 D1	15-06-2000
			DE 69608240 T2	23-11-2000
			EP 0790262 A1	20-08-1997
			JP 3231332 B2	19-11-2001
			US 6005034 A	21-12-1999
			CA 2203876 A1	06-03-1997
			WO 9708218 A1	06-03-1997
			TW 416962 B	01-01-2001
EP 0569860	A	18-11-1993	US 5366786 A	22-11-1994
			AU 3850793 A	18-11-1993
			CA 2080084 A1	16-11-1993
			DE 69312387 D1	04-09-1997
			DE 69312387 T2	06-11-1997
			EP 0569860 A1	18-11-1993
			ES 2106224 T3	01-11-1997
			JP 6049761 A	22-02-1994
			KR 236746 B1	02-03-2000
			MX 9302491 A1	01-12-1993
			US 5798167 A	25-08-1998
US 5137973	A	11-08-1992	NONE	